N72 31118

NASA CR-112127

FEASIBILITY DEMONSTRATION OF A HYPERFILTRATION TECHNIQUE TO RECLAIM SHOWER WASTEWATER AT ELEVATED TEMPERATURES

Prepared under Contract No. NASI-11297 by
College of Engineering
Clemson University
Clemson, South Carolina

for

National Aeronautics and Space Administration

For sale by the Office of Technical Services, Department of Commerce:
Washington, D. C. 20230 -- Price: $0.75

Foreword

This document was prepared by the College of Engineering, Department of Mechanical Engineering, Clemson University, for the Langley Research Center of the National Aeronautics and Space Administration. This effort was administered under the technical direction of Mr. John B. Hall, Jr.

The cooperation of Dr. John Andrews of Environmental Systems Engineering at Clemson University, of Dr. Malcolm Paynter of the Microbiology Department at Clemson, of Dr. Norwood Page of the Clemson Chemical Services Laboratory, and the technical support of the Oak Ridge National Laboratory are hereby acknowledged. Without this support the program could never have been conducted.

Principal investigators of this program were Drs. J. C. Hester and C. A. Brandon and valuable technical assistance was provided by Professor J. K. Johnson.

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SUMMARY

A feasibility demonstration of a hyperfiltration technique to determine its capability to reclaim shower wastewater at elevated temperature was conducted by Clemson University. Approximately twenty (20) gallons of typical shower water were processed through a dynamically formed membrane at a temperature of 167°F. Chemical and bacterial analyses of the product water are presented which show compliance with all potable water requirements established for extended manned space missions. In addition, subsystem characteristics and capabilities are discussed.

INTRODUCTION

To date, hyperfiltration water reclamation subsystems under development by NASA have been somewhat limited by subsystems requirements of simplicity and operation at elevated temperatures to control micro-organisms in the recovered water without the use of biocides. Clemson University has been using advanced hyperfiltration techniques in textile waste applications at elevated temperatures and it appears logical to extend this technology to spacecraft washwater applications. It was established that twenty (20) gallons of representative showerwater would be processed at a temperature of 165°F to determine the feasibility of the hyperfiltration technique to produce potable water.

This report contains the results of the program which was performed to arrive solely at a feasibility position. It was not intended to derive design parameters during the effort.

FEASIBILITY DEMONSTRATION

The following sections describe the procedures and results obtained during this program.

Test System

Figure 1 shows a schematic of the test configuration. Two loops of this configuration were constructed. The first configuration consisted of cast iron piping, etc. and was used solely in subsystem orientation, etc. The actual test configuration was constructed of stainless steel and vinyl tubing as shown in Figure 2. These materials were selected in order to maintain minimal interaction between subsystem materials and the waste water inputs. The chosen test section, shown schematically in Figure 3, consisted of a seven channel ceramic tube with average pore size of 0.27 microns. This tube was used for membrane support (see Figure 4) and an annular chamber was provided around the tube to collect the product water. Product flux was radially outward through the ceramic tube.

The test section was approximately fourteen (14) inches long with an available cross-sectional flow area of .0379 in^2. This design provided a membrane area of 0.178 ft^2 per tube. Other support configurations including porous ceramic, carbon, and metallic tubes of various cross sections and lengths could be used for this application. The seven channel ceramic tube was chosen because of its availability and the existence of a proven test section design.

The primary difficulty in subsystem design involved the availability of a high pressure, high temperature, stainless steel pump. The pump used for this investigation was obtained from Goulds Pumps Incorporated, Model MP3913. The pump was not optimally sized for this application (10 gallons

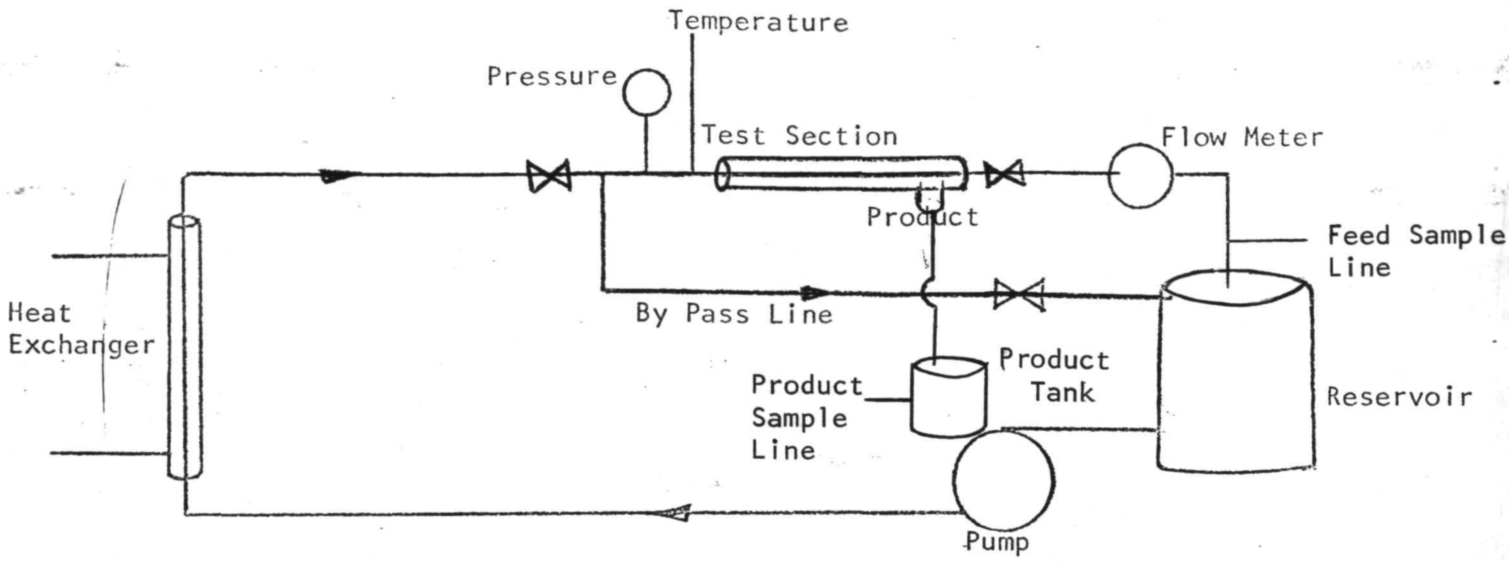


Figure 1 - Test Configuration Schematic

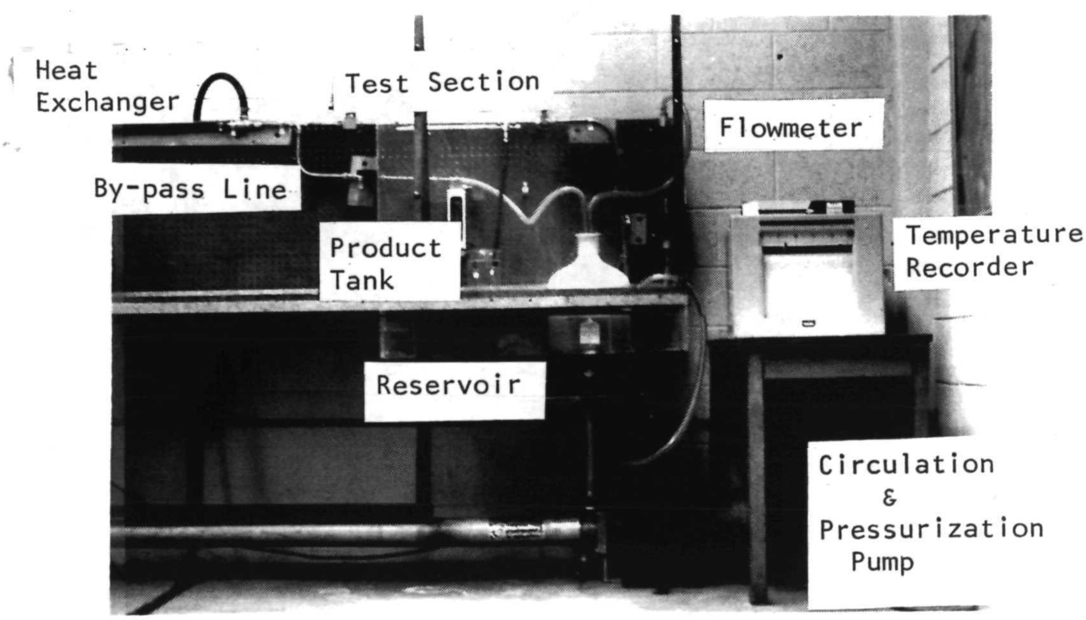


Figure 2 - Test Configuration Photograph

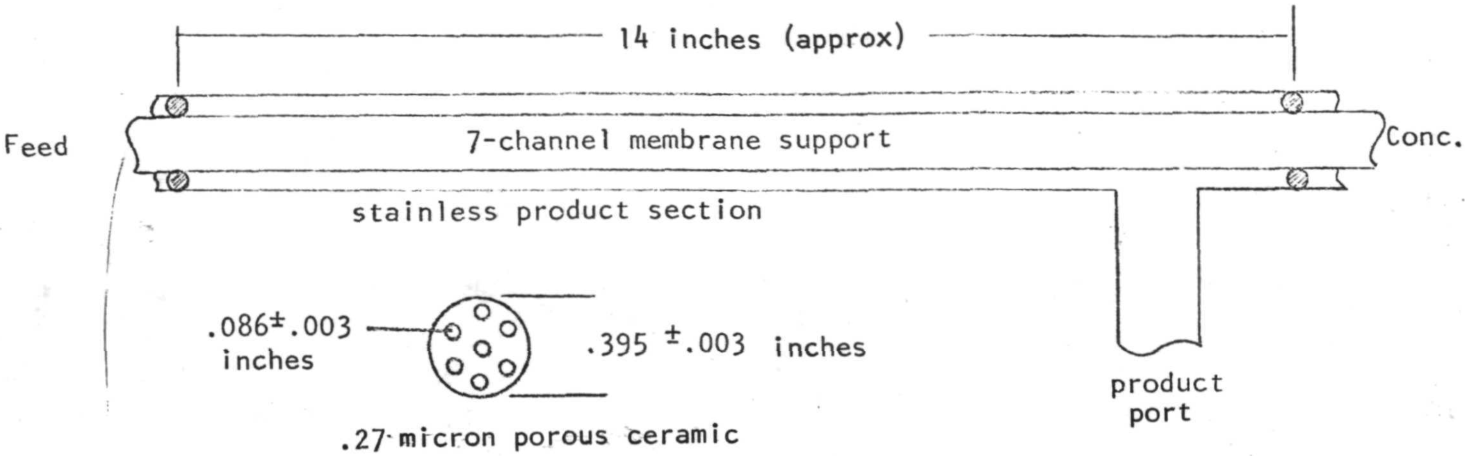


Figure 3 - Membrane Support - Test Section Schematic

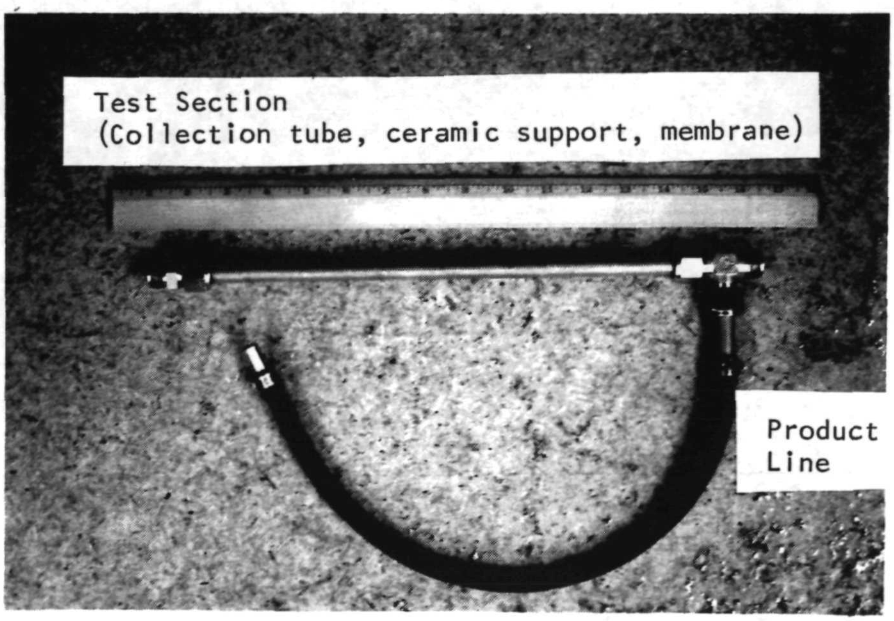


Figure 4 - Membrane Support - Test Section Photograph

per minute at high pressures), but it was selected due to its availability to meet the program schedule.

Membrane Formation

The membrane used in the hyperfiltration demonstration was a dual-layer, dynamically-formed matrix consisting of a hydrous Zr(IV) oxide layer covered with a polyacrylic acid layer. The membrane was formed as follows:

1) A NaOH solution (1M) was circulated through the loop for an hour at 350 psig.

2) The NaOH was rinsed out with distilled water. Then a 1 M HNO_3 solution was circulated through the loop for an hour under a pressure of 350 psig.

3) The loop was then rinsed with distilled water for a minimum of thirty minutes. During the final water rinse, care was taken to flush residual solution from auxiliary lines and valves since minute amounts of certain impurities may interfere with membrane formation.

4) A solution of 0.05 M NaCl and 10^{-4} M hydrous Zr(IV) oxide was adjusted with HCl to a pH of 4 and introduced into the feed tank.

5) Circulation velocities and pressures were adjusted to typical values of 25-30 ft/sec and 700-900 psig.

6) NaCl rejection was monitored until it reached 40-50%. (This took from two to four hours.) At this time the system pH was adjusted to 2-2.3 with HCl and then a solution of 50 ppm polyacrylic acid was added.

7) The acid feed was circulated for 25-30 minutes and then the pH was adjusted to approximately 3.0 by the addition of 1 M NaOH.

8) An additional 30-minute circulation was followed by another pH adjustment of one pH unit. Incremental increases in pH were then repeated until the pH was 6.5-7.0. At this time the system was rinsed with distilled water, and the membrane was considered formed.

Feasibility Test

The feasibility test consisted of batch processing of twenty-one (21) gallons of representative shower water. The shower water was accumulated by taking individual showers each consisting of one gallon of distilled water and 10 ml of Miranol (C2M-SF) soap. The shower water was collected in 5 gallon containers from various donors, then strained through cheese cloth and stored at 40°F until required for processing. Initial loop volume was 3 gallons of distilled water at test initiation. The system was then loaded with cold shower water and a sample taken. The shower water was raised to a temperature of approximately 167°F and maintained at this temperature for the remainder of the test although there were temperature excursions as high as 175°F. Samples of feed and product water were taken approximately every two hours for Clemson University tests (CUT) and at initial, mid, and final process intervals for NASA analyses. Results are given in the following section.

Clemson University Test (CUT) Results

Chemical and bacterial analyses of both the product and the feed water were conducted by Clemson University personnel. The bacteria tests consisted of 48 hour growths on trypticsoy agar. Standard chemical lab practices were

used for the chemical analyses. The results are given in Tables I and II. Table III summarizes the rejection characteristics of the membrane unit.

NASA Results

Parallel chemical and bacteria analyses were conducted by NASA Langley Research Center personnel to verify Clemson University data and to obtain complete ad hoc contaminant results. The results of these analyses are presented in Tables IV and V.

The NASA results, taken at initial, mid, and final test conditions show excellent compliance with ad hoc requirements. The only exception to total compliance with these ad hoc requirements is in ammonia where the final product had 1.2 ppm as compared to an ad hoc requirement* of 1.0.

*Space Science Board: Report of the Ad Hoc Panel on Water Quality Standards for Long Derivation Manned Space Missions. Nat. Acad. Sci.-Nat. Res. Counc., Sept., 1967.

TABLE I

CLEMSON UNIVERSITY CHEMICALL ANALYSES

Sample Ident.	Type*	Time**	NH_4	Urea	Cl	K	Na	SO_4	pH	Alkalinity (as $CaCO_3$)	Specific Cond (μmhos/cm)	COD	Total Solids	Filterable Solids
C1	F	1715	1.7	20.0	13.5	42.0	84.0	1.16	8.21	21.0	361.1	1666.9	824.	77.0
C2	P	1715	<1.0	18.4	2.1	2.4	6.6	0.68	8.34	2.9	31.5	99.1	150.	9.0
C3	F	2030	2.7	22.3	21.3	55.0	93.0	3.88	7.69	25.8	442.1	2178.4	1144.	206.
C4	P	2030	<1.0	21.4	3.5	3.3	7.1	0.78	9.18	4.1	38.3	101.79	234.	1.0
C5	F	2300	3.1	21.8	25.2	75.0	83.0	3.40	7.99	29.9	497.6	2454.3	1288.	214.
C6	P	2300	<1.0	20.6	2.8	4.5	6.3	1.26	7.29	3.2	29.2	91.0	206.	3.0
C7	F	0100	5.0	18.8	16.3	89.0	95.0	4.55	8.04	34.0	576.5	2880.1	1570.	208.
C8	P	0100	<1.0	21.5	2.1	5.3	7.5	1.55	7.23	4.1	46.3	96.2	198.	6.0
C9	F	0330	4.8	22.7	36.6	107.0	115.0	9.20	8.24	40.6	707.0	3351.8	1940.	201.
C10	P	0330	<1.0	23.1	2.8	6.0	8.2	1.74	7.06	4.2	52.0	101.8	212.	7.0
C11	F	0500	5.8	21.8	40.5	142.0	135.0	13.6	8.22	50.0	858.7	2094.3	2260.	246.
C12	P	0500	<1.0	24.0	3.5	7.7	10.1	1.45	9.46	3.1	61.0	113.3	192.	6.0
C13	F	0600	4.6	26.4	56.4	156.0	156.0	18.1	8.25	52.2	983.1	4920.8	2496.	296.
C14	P	0600	<1.0	23.1	4.3	8.3	10.6	1.74	7.42	5.1	66.5	111.8	190.	11.0

*F = Feed
P = Product

**τ = 0 @ 1415

TABLE II

CLEMSON UNIVERSITY BACTERIA ANALYSES

Sample Number	Time From Initial Sample	Colony Count* 48-hour	Remarks
B1	0	1-300/ml	Feed after holding at >165°F for 1.0 hour
B2	1:40	0/100 ml	Product
B3	5:25	0/100 ml	Product
B4	8:11	0/100 ml	Product
B5	10:06	0/100 ml	Product
B6	11:40	0/100 ml	Product
B7	14:37	0/100 ml	Product

*Ad Hoc Spec < 10/ml

TABLE III

MEMBRANE REJECTION CHARACTERISTICS

Sample Part	Type	*Rejection(Percent)						
		Cl	K	Na	Specific Conductivity	COD	Total Solids	Filterable Solids
1 2	F P	84.5	94.3	92.1	91.3	94.1	91.8	89.3
1 2	F P	83.6	94.0	92.4	91.5	95.4	89.6	99.5
1 2	F P	89.9	94.0	96.9	92.1	96.3	84.1	98.6
1 2	F P	87.1	94.0	92.1	94.0	96.7	87.4	96.1
1 2	F P	92.3	94.0	92.9	92.6	96.9	89.1	96.5
1 2	F P	91.4	94.6	92.5	92.9	96.3	91.5	97.5
1 2	F P	92.4	94.7	93.2	93.4	97.7	92.4	96.3

$$^*\text{Rejection} = 1 - \frac{\text{Concentration Product}}{\text{Concentration Feed}}$$

F = Feed

P = Product

TABLE IV

NASA CHEMICAL ANALYSES

Contaminant	Maximum Allowable Concentration NAS-SSB *ad hoc* Panel	Units	Results Feed Cold	Feed Initial	Feed Mid	Feed Final	Product Initial	Product Mid	Product Final
Arsenic	0.5	ppm	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.01
Barium	2.0	"	<1	<1	<1	<1	<1	<1	<1
Boron	5.0	"	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Cadmium	0.05	"	0.01	0.01	0.04	0.05	0.01	0.005	0.025
Chromium	0.05	"	0.41	0.18	0.01	0.03	0.01	0.01	0.01
Copper	3.0	"	<0.1	0.3	0.2	0.5	<0.1	<0.1	<0.1
Iron	*	"	---	---	---	---	---	---	---
Lead	0.2	"	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Manganese	*	"	---	---	---	---	---	---	---
Selenium	0.05	"	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Silver	0.5	"	<0.05	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zinc	*	"	---	---	---	---	---	---	---
Ammonia	1.0	"	7.0	3.4	8.0	11	0.4	0.8	1.2
Chloride	450	"	52	82	120	112	<5	5	11
Cyanide	*	"	---	---	---	---	---	---	---
Fluoride	2.0	"	0.2	0.1	0.2	0.2	0.2	0.2	0.2
Nitrates, Nitrites	10.0	"	<0.5	1.9	2.0	3.0	<0.5	<0.5	0.5
Sulfate	250	"	50	10	35	110	5	5	5
Alkyl Benzene Sulfonate	No foaming	"	<0.1	<0.1	<0.1	<0.1	<0.1	<0.01	<0.01
Carbon Chloroform Ext.	*	"	---	---	---	---	---	---	---
Phenols	*	"	---	---	---	---	---	---	---
Organic Carbon	*	"	1200	720	1200	2000	43	44	56

TABLE IV - cont'd.

Contaminant	Maximum Allowable Concentration NAS-SSB *ad hoc* Panel	Units	Results						
			Feed				Product		
			Cold	Initial	Mid	Final	Initial	Mid	Final
Urea	*	ppm	<50	<50	<50	<50	<50	<50	<50
Color ($PtCl_6$ Units)	15	$PtCl_6$ Units	>100	>100	>100	>100	<5	<5	<5
Conductivity	*	10^{-6} mhos/cm	380	290	440	850	31	44	62
Odor	*	ppm	Soapy	None	Soapy	Soapy	None	None	None
pH	*	"	7.4	8.0	8.6	8.6	9.5	9.6	9.7
Total Solids	*	"	1600	400	1400	2500	<100	<100	<100
Turbidity (ppm Silica)	10 ppm Silica	"	55	34	80	150	9	8	9.0

TABLE V

NASA BACTERIA ANALYSES

Sample	Time from Initial Sample	Colony Count*	Remarks
NASA-1	0	1.35×10^7/ml	Cold Shower Water-Baseline
-2	1:55	2.0×10^0/ml	Initial Feed (after 1.0 hr>165°F)
-3	1:55	1.0×10^{-2}/ml	Initial Product
-4	9:56	$<1.0 \times 10^{-2}$/ml	Midpoint Feed
-5	9:56	$<1.0 \times 10^{-2}$/ml	Midpoint Product
-6	16:30	$<1.0 \times 10^{-2}$/ml	Final Feed
-7	16:30	$<1.0 \times 10^{-2}$/ml	Final Product

Ad Hoc Spec: <10/ml

SYSTEM CHARACTERISTICS

The following sections detail specific characteristics of the subsystem and address subsystem capability in washwater reclamation for a 180-day resupply mission with system requirements of 50 gallons of washwater per day. The results are presented as general data and are not intended as specific design data.

Process Rates

Process flux rates (gallons/ft^2 day) vary from membrane to membrane and are directly influenced by system pressure and temperature; as both increase so does the flux increase. The variation is approximately linear with pressure and varies directly with the viscosity variation with temperature. At temperatures of approximately 165°F and pressures of 700-900 psia a flux level of 150-250 gallons/ ft^2 day is anticipated. For this particular feasibility demonstration a level of 180 gallons/ft^2 day was achieved at the beginning of the test and the level degraded only about 5% during the conduct of the program. Thus, for the 50 gallon per day requirement a membrane area of approximately 0.3 ft^2 would be required. This could be met with a pair of test sections of the type presented in Figure 3. For alternative support structures, carbon or porous metal, for example, the flux rates will vary from those given here and empirical data will be required to arrive at these values.

The process rates quoted here are based upon representative contaminant levels in the washwater. No prefiltration is required for this subsystem and anything (food scraps, etc.) that will pass the pump and is smaller than the primary flow passages will not degrade membrane performance. This is a distinct advantage of this subsystem as compared to other competing techniques which require extensive pre-filtration.

Rejection Efficiency

It appears practical from a review of Tables II, III, and IV to design systems with a rejection efficiency of 85-96% for the constituents of typical washwater except urea. However, since the system exhibits approximately a 10:1 decrease in ammonia levels, the urea could be degraded to ammonia and handled safely. All other constituents either meet or exceed the ad hoc committee requirements for potable water. The rejection efficiency is essentially independent of operating pressures and temperatures.

Recovery Efficiency

The demonstrated recovery efficiency of the subject concept was only 81%, but this does not represent a system design value because no attempt was made to maximize this figure. This value merely represents the water out/water in ratio for the minimum pumping volume of one particular system. Future work should attempt to attain true recovery efficiency values by eliminating the minimum pumping volume as the limiting variable. No calculations were performed using ad hoc requirements and observed rejection characteristics to obtain theoretical recovery efficiencies since it is not known if the rejection characteristics hold for an extreme range of concentration levels. This question should be answered by future development tests.

Power Requirements

Power is required for the subject concept for circulation, pressurization, initial heating, and temperature maintenance. The particular level of each parameter, and the particular subsystem design involved (flow through vs. single pass), determines the power requirements, but general guidelines can be given.

Circulation velocities of 8-35 ft/sec over the membrane are required to preclude fouling and system pressures of 350-1200 psig are anticipated, as dictated by available membrane area and desired flux levels. System temperatures will certainly exceed 165°F, but the subsystem is capable of operating at even higher temperatures should the design dictate. Initial heating rates will be determined by minimum subsystem response requirements.

The subsystem used in the feasibility demonstration was oversized in order to obtain a wide range of operating variables. However, sufficient experience has been gained with the subsystem to allow reasonable projections of operational subsystem power requirements for a typical system.

For 50 gallons/day, it would only be necessary to have approximately 0.3 ft^2 of membrane area since flux levels of 150-180 gallons/ft^2 day at elevated temperatures are anticipated. For a typical 7-channel module velocities of 30 ft/sec are obtained at flow rates of 3.6 gpm. Such velocities are sufficient to preclude fouling, etc. of the membrane. Coupling this requirement with the operational pressures dictates the power requirements. System operational pressures range from 350 to 1200 psig.

The more efficient means of meeting these conditions is through a pressurization pump operating in tandum with a circulating pump. The pressurization pump would expend energy only on make-up water and the circulation pump would overcome pumping losses only. Assuming the configuration indicated above the pressurization pump would be only a fractional hp type since the flow rate would be only 50 gallons/day while the circulation pump would require approximately 90 watts since a Δp of only 30 psi is involved. The alternative approach is to use a single pump for both circulation and pressurization. This results in a power requirement given by

$$\dot{p} = \dot{m}\, \nu\, dp/\varepsilon$$

$$= \left(\frac{29.3\ \text{lbm}}{\text{min}}\right) \left(\frac{\text{ft}^3}{60.8\ \text{lbm}}\right) \left(\frac{1000\ \text{lb}_f}{\text{in}^2}\right)/.58$$

$$\dot{P} = 152.0\ \frac{\text{Btu}}{\text{min}} = 3.6\ \text{HP} = 2.67\ \text{kw}$$

where $\dot{m}$ = mass flow rate

ν = specific volume

ε = pump efficiency

System Weight Projections

The inherent simplicity of the subject concept leads directly to a light weight system. A representative system is presented in Figure 5. Exclusive of instrumentation and pump the system should weigh less than four (4) pounds. Including pump and instrumentation, a weight of forty pounds appears reasonable.

Replacement and Repair Considerations

There are two distinct methods that may be used in subsystem repair. The first, and least attractive, involves in situ reformation of the membrane by the procedure discussed in the membrane formation on page 6. For certain types of missions, including long term, minimum weight, etc., this procedure may have merit, but the better approach for general missions involves the use of preformed membranes. The weight of the replacement test section would be approximately one pound. Sealed test section assemblies, stored wet, would be carried as spares on board and shuttled via resupply missions as need dictated.

Microbiological Factors

A review of the biological analyses conducted by NASA and Clemson University indicates that the 165°F exposure and the subject membrane configuration

are satisfactory in preventing micro-organism build-up. Test data show no colonies in the product water at any time, even when the feed is highly contaminated. Data also show that prolonged exposure to the 165°F environment kills the micro-organisms even in the feed. However, the data indicate that 1.0 hour at this temperature is not satisfactory for this purpose. If higher temperatures are required, then membrane appears compatible with this condition. Thus, it appears totally practical to consider a subsystem for washwater treatment which does not include the use of a biocide in the feed.

Membrane Lifetime

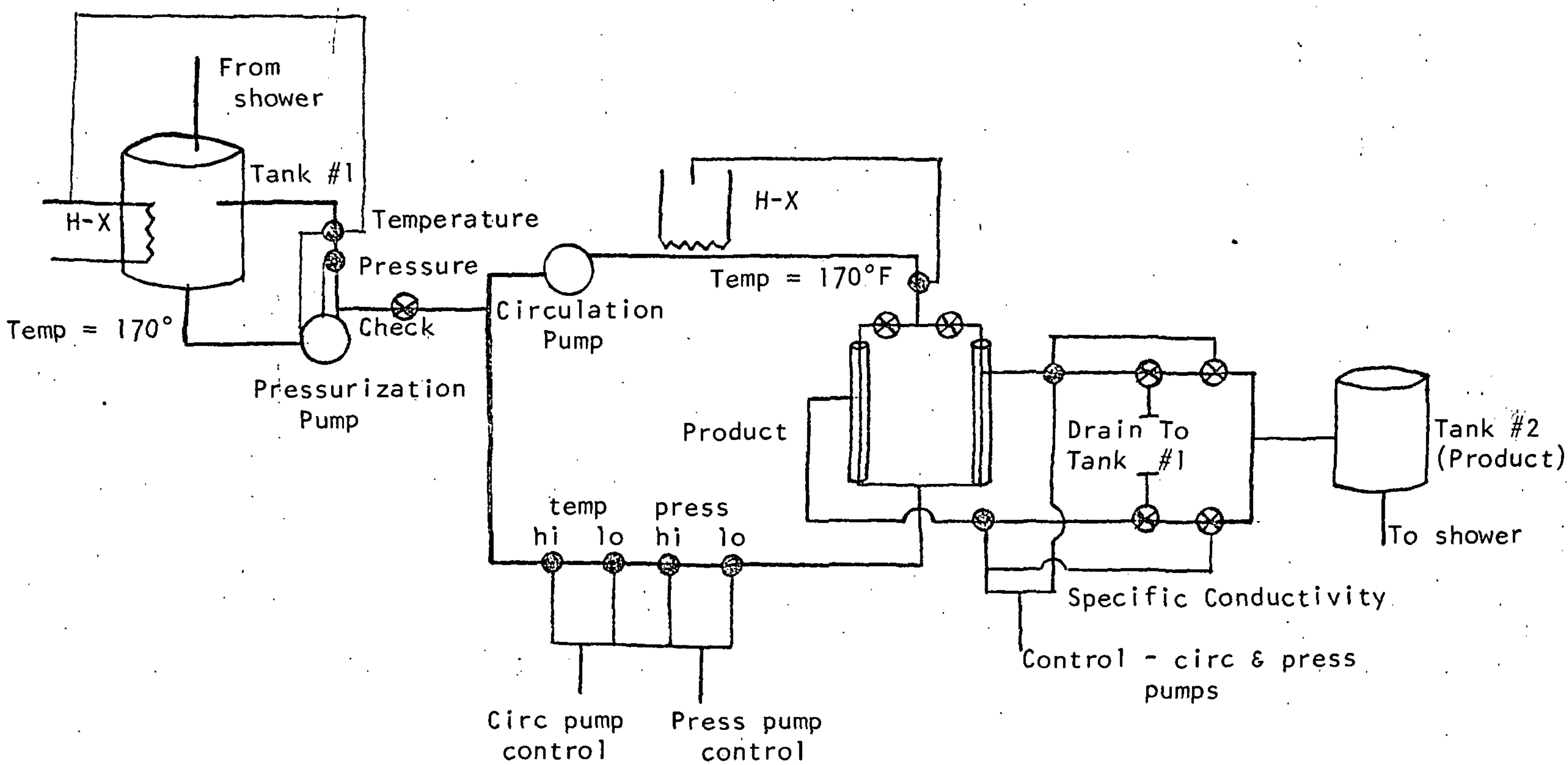
Obviously membrane life determination was not a portion of the feasibility demonstration. However, it is an extremely important variable and must be determined prior to actual system design. No data exists to indicate lifetime in washwater applications or in applications where sterility is important. Test data, using representative duty cycles, must provide this information. However, data has been generated in textile waste treatment to indicate membrane lifetimes in excess of 2000-3000 hours. The acquisition of degradation and life data should be addressed thoroughly in future development activities.

CONCLUSIONS AND RECOMMENDATIONS

The dynamically formed hyperfiltration concept has been demonstrated as being capable of processing representative shower water at temperatures in excess of 165°F while satisfying all NASA ad hoc panel requirements for the processed water. Pertinent test findings include:

1) Continuous operation at $>$165°F and the complete elimination of all viable micro-organisms in the product.
2) Flux levels of 180 gallons/ ft^2·day obtained at 167°F.
3) Rejection efficiencies for various constituents ranging from 85-96%.
4) Low power and weight requirements
5) Good recovery efficiency (although exact level not a program goal)
6) Inherent simplicity and reliability

Based upon the test results it is recommended that (a) lifetime and recovery efficiency data be obtained, and (b) a prototypical unit similar to the configuration of Figure 5 be developed to obtain basic design data.



BASELINE CONFIGURATION

Figure 5

CLEMSON UNIVERSITY

CLEMSON, SOUTH CAROLINA 29631

COLLEGE OF ENGINEERING

DEPARTMENT OF
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TELEPHONE 656-3470
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September 21, 1972

Mr. B. C. Baccus
Contracting Officer
National Aeronautics and
Space Administration
Langley Research Center
Hampton, Virginia 23365

Contract NAS 1-11297

Dear Mr. Baccus:

As of September 21, 1972 the final reports relative to the subject contract have been distributed consistent with your requirements issued August 11. This completes our obligation with respect to the subject contract. We feel that the work performed has been of a mutually beneficial nature and we look forward to working with your center in future activities. Should you have any unanswered questions relative to the study, please do not hesitate to contact me.

Sincerely,

J. Charles Hester
Principal Investigator

JCH/mae

cc: Dean S. F. Hulbert
Mr. A. L. McCracken
Mr. M. A. Wilson